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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

THOMAS DANIEL, ET AL. :

: EXAMINER: METZMAIER, D. S.

SERIAL NO: 09/831,915 :

FILED: MAY 25, 2001 :

: GROUP ART UNIT: 1712

FOR: HYDROGELS CAPABLE OF  
ABSORBING AQUEOUS FLUIDS :DECLARATION UNDER 37 C.F.R. § 1.132COMMISSIONER FOR PATENTS,  
ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Dr. Manfred Essig who deposes and states:

1. That I am a graduate of Universität Kaiserslautern and received a Ph.D. degree in the year 1981.
2. That I have been employed by BASF AG, for 18 years as a scientist in the field of Electron Microscopy.
3. That the following experiments were carried out by me or under my direct supervision and control.

The dried hydrogel according to the present invention was investigated by Scanning Electron Microscopy (SEM) to show that the silicon in the dried hydrogel particles is distributed throughout their bulk and not just on the surface of the particles.

In preparation of the measurement, the hydrogel particles were fixed in a polymer block. Then the polymer block was cut through by means of high-speed rotating diamond cutting tool to prepare cross sections of the dried hydrogel particles.

The SEM method performed is a well known standard technique described in the attached pages from the BASF internal website which are incorporated by reference into the Declaration. The SEM method allows the analysis of the distribution of elements on the dried hydrogel particles.

The sheet of three micrographs depicting the identical field of view, attached herewith, is incorporated into the Declaration by reference. On this sheet, the first image from the top, a Backscattered Electron Image, shows the cross section of particles without reference to a particular element. The second image from the top shows the elemental distribution of sodium and the third image shows the elemental distribution of silicon in the cross section of the particles. Clearly, the third image, in comparison with the first image, shows that silicon is present throughout the particle and not just on the surface.

In addition the micrographs elucidate the silicon distribution in the bulk of the particles as being caused by kneading ending up with a marble like phase separation.

4. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.

5. Further deponent saith not.

  
\_\_\_\_\_  
Signature

05/06/05  
\_\_\_\_\_  
Date

## Scanning Electron Microscopy (SEM)

### Contact

### Principle

In SEM a focused beam of electrons about 3 nm in diameter scans across the surface of the sample. The signal thus obtained depends on the SEM operating mode. It is detected synchronously to the position of the scanning electron beam and recorded in an image-storage device.

Differences in the topography of a sample give rise to differences in the intensity of the signals formed by the secondary electrons in the detector resulting in an apparently three-dimensional image that is typical of SEM (Fig. 1).

### Applications

An advantage of SEM over TEM is the ease of preparing the samples. As a rule, sputter deposition of a thin conductive gold layer on the surface of the sample suffices. Specimens of several cm in size can be examined at magnifications of 10x for a general view, up to  $10^5$ x for a detailed view. Special devices, e.g. a tensile tester, allow in-situ stress-strain experiments to be observed under the microscope at magnifications of up to 5000x. In common with TEM, SEM allows EDX spectra to be recorded and presented in the form of element distribution maps (Fig. 2). Systems containing water, e.g. dispersions or biological specimens, and substances with a low melting point, e.g. wax crystals, can be fixed by shock freezing, cryotransfer into the SEM, and direct imaging at low temperatures (Fig. 3).

Another means of examining samples containing water is possible with an environmental SEM (ESEM). The instrument is equipped with a special detector and a multistage pump system that permits microscopic investigation at vacuums down to 50 Torr without sputtering. Dissolving, crystallization, and film-formation mechanisms as well as biological samples can thus be studied without dehydration.

A disadvantage of SEM compared with TEM is that imaging is restricted to surfaces, and the magnification and

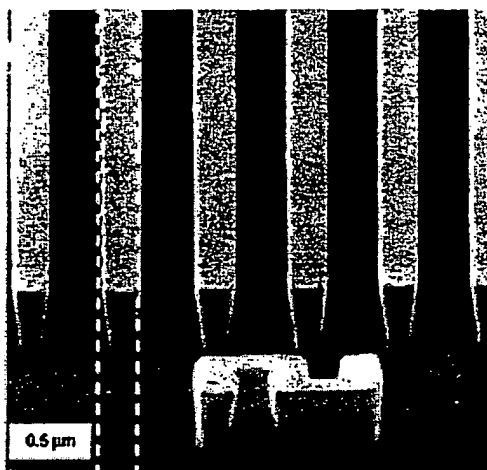


Fig. 1: SEM image of a structured microresist finish

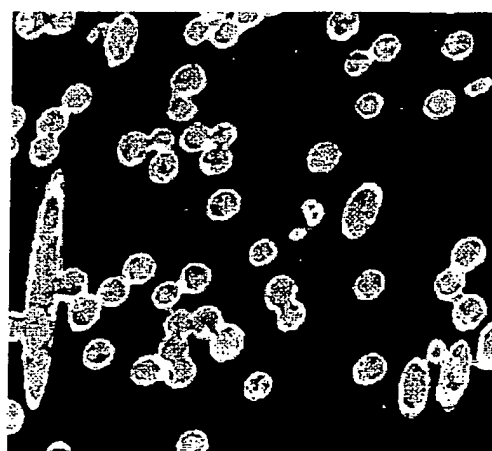
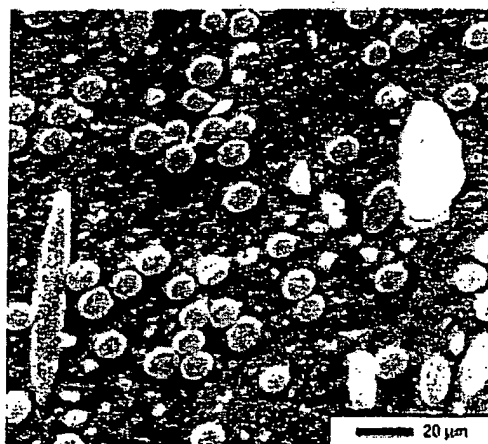


Fig. 2: Ground and polished section of a glass-fiber-reinforced polyamide containing a red phosphorus flame retardant. SEM image with corresponding Si (yellow/green)/P (red) distribution map



SEM

resolution are less.



Fig. 3: Growth of fungi on wheat leaf



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**Literature**

L. Reimer, G. Pfefferkorn, Raster-Elektronenmikroskopie, Springer-Verlag, Berlin, Heidelberg, 1973

D.B. Williams, J.I. Goldstein, C.E. Fiori, Principles of X-Ray Energy-Dispersive Spectrometry in the Analytical Electron Microscope in Principles of Analytical Electron Microscopy, Eds. D.C. Joy, A.D. Romig, Jr. and J.I. Goldstein, Plenum Press, New York, 1986

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GKP Home

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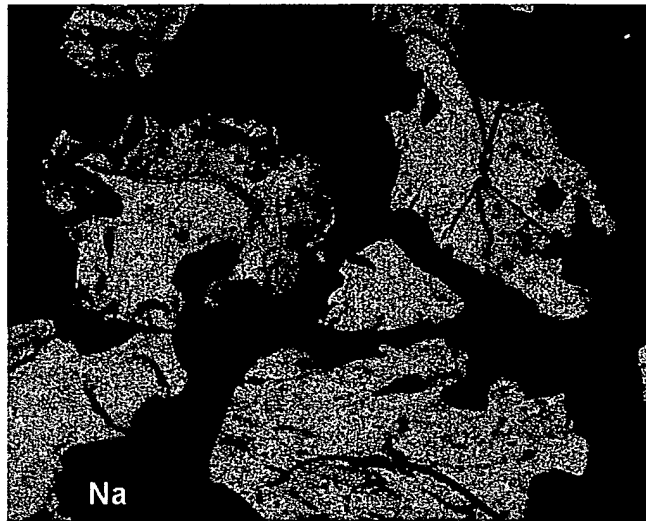


100 : 1

200µm

Auftrag: 49457

Abb:01e 100 RE



Auftrag: 49457

Abb:01 Na



Auftrag: 49457

Abb:01 Si